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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
09/806,274	03/27/2001	Wayne Edward Beimesch	390780)	6754
7*	590 05/06/2004		EXAM	INER
Peter C Knops			ROGERS, DAVID A	
Lathrop & Gage 2345 Grand Boulevard Suite 2800			ART UNIT	PAPER NUMBER
Kansas City, N			2856	
•			DATE MAILED: 05/06/200	14

Please find below and/or attached an Office communication concerning this application or proceeding.

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	Application No.	Applicant(s)
r · c	09/806,274	BEIMESCH, WAYNE EDWARD
Office Action Summary	Examiner	Art Unit
	David A. Rogers	2856
The MAILING DATE of this communication a riod for Reply		
A SHORTENED STATUTORY PERIOD FOR RESTHE MAILING DATE OF THIS COMMUNICATION Extensions of time may be available under the provisions of 37 CFR after SIX (6) MONTHS from the mailing date of this communication. If the period for reply specified above is less than thirty (30) days, a If NO period for reply is specified above, the maximum statutory period for the specified above is less than thirty (30) days, a If NO period for reply within the set or extended period for reply will, by statutory period for the specified above.	N. 1.136(a). In no event, however, may be reply within the statutory minimum of the iod will apply and will expire SIX (6) Movement to be some the application to be some the property of the same the application to be some	reply be timely filed irty (30) days will be considered timely. NTHS from the mailing date of this communication. ARANDONED (35 U.S.C. § 133).
atus		
1) Responsive to communication(s) filed on 19	9 March 2004.	
2a) ☐ This action is FINAL . 2b) ☑ 1	This action is non-final.	ottors, prospection as to the merits is
3) Since this application is in condition for allo	wance except for formal marks of Expanse Quaylo 1935 C	D 11 453 O.G. 213.
closed in accordance with the practice und	er Ex parte Quayle, 1905 C	.5. 11, 100 0.0. 211
isposition of Claims		
4) ⊠ Claim(s) 1-10 is/are pending in the applicate 4a) Of the above claim(s) is/are with 5) □ Claim(s) is/are allowed. 6) ⊠ Claim(s) 1-10 is/are rejected. 7) □ Claim(s) is/are objected to. 8) □ Claim(s) are subject to restriction and	drawn from consideration.	
Application Papers		
9) The specification is objected to by the Example 10) The drawing(s) filed on 27 March 2001 is/a Applicant may not request that any objection to Replacement drawing sheet(s) including the ∞	are: a)⊠ accepted or b)⊔ o the drawing(s) be held in abe orrection is required if the drav	yance. See 37 CFR 1.00(a). ring(s) is objected to. See 37 CFR 1.121(d)
11) The oath or declaration is objected to by the	ne Examiner. Note the attac	
Priority under 35 U.S.C. § 119		
11) The oath or declaration is objected to by the Priority under 35 U.S.C. § 119 12) Acknowledgment is made of a claim for for a) All b) Some * c) None of: 1. Certified copies of the priority docuted to 2. Certified copies of the priority docuted application from the International Between the standard detailed Office action for the standard to be supplied to be	reign priority under 35 U.S. ments have been received ments have been received priority documents have bureau (PCT Rule 17.2(a)).	C. § 119(a)-(d) or (f). in Application No een received in this National Stage
Priority under 35 U.S.C. § 119 12) Acknowledgment is made of a claim for fo a) All b) Some * c) None of: 1. Certified copies of the priority docu 2. Certified copies of the priority docu 3. Copies of the certified copies of the application from the International B	reign priority under 35 U.S. ments have been received, ments have been received a priority documents have be bureau (PCT Rule 17.2(a)). a list of the certified copies	C. § 119(a)-(d) or (f). in Application No een received in this National Stage

Application/Control Number: 09/806,274

Art Unit: 2856

DETAILED ACTION

- 1. In view of new references available prosecution of this application is hereby reopened. The Office apologizes for any inconvenience this may have caused.
- 2. The USPTO is in the process of moving to new office spaces in Alexandria, Virginia. Please note the new phone numbers for the examiner and the examiner's supervisor below.

Claim Rejections - 35 USC § 112

- 3. The following is a quotation of the second paragraph of 35 U.S.C. 112:

 The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.
- 4. Claim 1 recites the limitation "said *solid* material" in line 6. There is insufficient antecedent basis for this limitation in the claim.

Claim Rejections - 35 USC § 103

- 5. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:
 - (a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.
- 6. Claims 1, 3, 4, and 6 are rejected under 35 U.S.C. 103(a) as being unpatentable over United States Patent 5,140,845 to Robbins in view of "Chemical Principles" to Masterton *et al.* and "Compilation of Air Pollutant Emission Factors, AP-42" to the Environmental Protection Agency (EPA).

Application/Control Number: 09/806,274

Art Unit: 2856

Robbins teaches a method and apparatus for measuring volatile organic compounds (VOCs) in soils. In particular, Robbins teaches a resealable polyethylene bag (reference item 120) into which is placed a sample (an aqueous sample or a soil sample mixed with distilled water) (column 3, lines 35-37). After sealing, the bag is agitated to promote the release of any VOCs present in the sample. Robbins further reiterates what is already well-known in the art by citing from "Water Quality" to Tchobanoglous. This citation, generally on column 2, lines 55-68 and column 3, lines 1-7, teaches that time, mass size, and temperature all directly affect headspace equilibrium. At any rate, Robbins teaches that the headspace in the sealed bag is to reach equilibrium prior to testing for VOCs. In testing for VOCs, Robbins teaches that a flame ionization detector (FID) is used as the means to detect the VOCs in the headspace. It is also known that gas chromatographs and FIDs can be used to determine analyte quantities in the sample based on the peak value, i.e. the measured response of the GC/FID.

Robbins teaches that storage tanks are a source of VOCs (column 1, lines 16-20). Robbins also teaches that it is beneficial to store the bag at an optimum temperature in order that the headspace reach a state of equilibrium (column 5, lines 1-6). Robbins also teaches that time is a relevant factor to reach the desired equilibrium in the headspace (column 4, lines 57-58). Finally, Robbins teaches that the initial mass of the sample is directly related to the measured equilibrium headspace concentration (equation 5, equation 7). Robbins does not expressly teach a method for testing materials from a process

Art Unit: 2856

system where the material is placed in a sealable bag and <u>is stored at the mean</u>

<u>exit temperature of said emissions of said system.</u>

Masterton et al. teaches the general scientific theory of liquid-vapor equilibrium in a closed system. A sealed flask is used in the example, but the scientific principles apply equally to a sealed bag. Masterton et al. teaches that a liquid placed in the closed system will, over time, reach a state of equilibrium with regard to the headspace. Equilibrium is the state wherein, at a given temperature, the number of molecules from the liquid entering into the vapor state (headspace) equals the number of molecules reentering the liquid state. As temperature increases the vapor pressure of liquids increase. Therefore, at higher temperatures a larger fraction of molecules will acquire enough energy to escape from the liquid to the vapor. This means that at higher temperatures more vapor molecules will be present in the headspace than at lower temperatures. Equilibrium, however, will be reached over time and will be independent of the temperature. It is just that more molecules will be present in the vapor when equilibrium is reached at higher temperatures thus increasing the probability of detection of the VOC molecules when the headspace is sampled.

Furthermore, the EPA continually promulgates information and guidance to the public regarding hazardous materials. Of particular relevance is EPA Method AP-42. Section 6.8 of this method, written July 1993, addresses soaps and detergents. Herein the Method AP-42 reiterates what the applicant already admits is well-known, and that is that certain processes such a spray drying

Application/Control Number: 09/806,274

Art Unit: 2856

release VOCs into the atmosphere. Furthermore, section 6.8.3.1 of Method AP-42 states (emphasis added)

The main atmospheric pollution problem in soap manufacturing is odor. The storage and handling of liquid ingredients (including sulfonic acids and salts) and sulfates are some of the sources of this odor. <u>Vent lines, vacuum exhausts, raw material and product storage, and waste streams are all potential odor sources</u>. Control of these odors may be achieved by scrubbing exhaust fumes and, if necessary, incinerating the remaining volatile organic compounds (VOC).

In section 6.8.3.2 it is stated (emphasis added):

In addition to particulate emissions, volatile organics may be emitted when the slurry contains organic materials with low vapor pressures. The VOCs originate primarily from the surfactants included in the slurry. <u>The amount vaporized depends on many variables such as tower temperature and the volatility of organics used in the slurry</u>. These vaporized organic materials condense in the tower exhaust airstream into droplets or particles. Paraffin alcohols and amides in the exhaust stream can result in a highly visible plume that persists after the condensed water vapor plume has dissipated.

Opacity and the organic emissions are influenced by granule temperature and moisture at the end of drying, temperature profiles in the dryer, and formulation of the slurry. A method for controlling visible emissions would be to remove offending organic compounds (i. e., by substitution) from the slurry. Otherwise, tower production rate may be reduced thereby reducing air inlet temperatures and exhaust temperatures. Lowering production rate will also reduce organic emissions.

The Method AP-42 is quite clear that the temperature of the process may, in fact, be a major causal factor in the release of VOCs into the atmosphere. Furthermore, drying towers, vent lines, vacuum exhausts, and waste streams are all regions within a process system whose temperature can be measured.

Art Unit: 2856

Section 7.1 of Method AP-42 details VOC emissions from systems such as storage tanks. This section also provides a listing of some known VOCs and their boiling points and vapor pressures (see table 7.1-3). This section also provides a detailed example on estimating the emission rate of VOCs from the material in a storage tank (see pages 7.1-73 through 7.1-83). This estimating process clearly shows that the *emission rate* is a function of the temperature of the material in the tank, the vapor space volume, and the equilibrium partial pressures of the volatile organic liquids in the material.

Replicating the conditions of a process used to create a product, e.g. replicating the temperature at which products are manufactured, would allow one of ordinary skill to determine if their process was indeed causing VOCs to be released into the atmosphere. Since vapor pressure of liquids increases with temperature, VOC release rates will be higher at points in the manufacturing process that are at elevated temperatures. These increased release rates must be monitored to ensure that the manufacturing process is in compliance with state and federal laws that require monitoring of VOCs (see also applicant's background of the invention). By understanding this basic relationship between temperature and vapor pressure one could then use techniques to minimize the amount of VOCs released, such as by scrubbing, incineration, substitution, and lower temperatures. See again section 6.8.3.1 of Method AP-42.

It is important to note that maintaining the closed system, i.e. the sealed bag, at the "mean exit temperature" of the process may be impractical. Bed

Art Unit: 2856

dryers and spray dryers can operate at very high temperatures that would cause the bag to melt (see Legros et al., cited below, where a dryer operates at 400 °C). And, since Masterton et al. teaches that headspace equilibrium will eventually be reached independently of the temperature, it will not be necessary to maintain the bag at such high temperatures. A higher temperature may be preferred since it will increase the vapor pressure of the material in the bag so that sufficient VOC molecules are released into the vapor phase thus increasing the probability of detection by a device such as an FID. Maintaining the temperature of the bag at an elevated temperature will also allow some facilities to determine if their specific process has a higher-thanallowed release rate of VOCs. It is also important to note that, with open systems such as spray dryers and fluid bed dryers, VOCs will inherently release into the atmosphere even if the exit temperature was low. This is because all liquids have a vapor pressure that is temperature dependent as noted above. A lower the temperature generally results in a lower emitting rate of a VOC. This can be seen in table 7.1-3 of the Method AP-42.

Robbins in view of Masterton *et al* and Method AP-42 teaches that it is known that, in a closed system, the initial mass and the temperature affect the equilibrium. The time to reach equilibrium is, therefore, dependent on amount of material, the temperature of the material, and the vapor pressure of the materials of interest. The time for reaching equilibrium, e.g. time between 5 and 24 hours, therefore, is dependent on knowing the conditions (temperature, sample size) being tested and would be determined on a case-by-case basis.

Art Unit: 2856

In summary, Robbins teaches that materials are placed in a sealed bag and maintained at a temperature until the headspace reaches equilibrium and then testing the headspace using an FID. Masterton et al. teaches the headspace equilibrium is reached independently of the maintained temperature, and that more molecules will be in the vapor state in the headspace at higher temperatures. Finally, Method AP-42 teaches that in a process system such as a drying tower the amount of VOCs released depends on the temperature of the tower. It would, therefore, have been obvious to one of ordinary skill in the art at the time of the invention to modify the teachings of Robbins with the teachings of Masterton et al. and Method AP-42 in order to provide a sample of material from a system, seal the material in a bag in order to have a headspace, and then to hold the material at a mean exit temperature of the emissions of the system in order to allow the headspace to come to equilibrium prior to testing for the presence of VOCs using techniques such as a flame ionization detector.

7. Claim 2 is rejected under 35 U.S.C. 103(a) as being unpatentable over Robbins in view of teachings of Master ton *et al.* and Method AP-42 as applied to claim 1 above, and further in view of United States Patent 5,809,664 to Legros *et al.*

Robbins in view the teachings of Masterton *et al.* and Method AP-42 show that it is known to test for VOCs using a sample stored in a sealed bag and where the source of the material can be a spray dryer or storage tank.

Application/Control Number: 09/806,274

Art Unit: 2856

Robbins in view the teachings of Masterton *et al.* and Method AP-42 does not expressly teach that the source of the material can be a fluid bed dryer.

Legros *et al.* teaches that it is known that fluid bed dryers are a source of VOCs and that incinerators are used to eliminate the VOCs released into the atmosphere (Abstract). Also, the applicant admits that "VOC measurement techniques have been developed and have been constantly employed to monitor VOC emissions of virtually every unit operation in every manufacturing facility throughout the world." Furthermore, by testing for VOCs in the material in the fluid bed dryer process, it can be determined if the incinerator is actually needed, i.e. the incinerator is turned off if the process shows that no VOCs are present.

It would have been obvious to one of ordinary skill in the art at the time of the invention to modify the teachings of Robbins in view of Masterton *et al.* and Method AP-42 so that a sample of material from the process of Legros *et al.* is tested in order to determine if the process is actually producing VOCs.

8. Claim 5 is rejected under 35 U.S.C. 103(a) as being unpatentable over Robbins in view of the teachings of Masterton *et al.* and Method AP-42 as applied to claim 1 above, and further in view of United States Patent 5,522,271 to Turriff *et al.* or Method 5035 to the EPA.

Robbins in view of the teachings of Masterton *et al.* and Method AP-42 teaches that it is known to place material in a sealed bag and to let the headspace in the bag reach equilibrium prior to sampling for the presence of VOCs. Furthermore, Robbins teaches that, in headspace sampling, a

Art Unit: 2856

consistent volume or weight of ground water or soil is placed in a container (column 1, lines 48-50) and the initial mass of the sample is critical to headspace equilibrium (columns 5-6). Robbins in view of the teachings of Masterton *et al.* and Method AP-42 does not expressly teach a sample size between 1 gram and 100 grams.

Turriff *et al.* teaches an apparatus for obtaining samples for VOC testing. The sampling device has a volume capacity of 25-30 grams (column 2, lines 64-67) and provides for the consistent volume for headspace sampling as required by Robbins. Also, Method 5035 was originally promulgated by the EPA in 1996. This method specifically teaches methods for testing of VOCs in solid materials such as soils, sediments, and solid wastes. This method can be used in conjunction with Method 8015 which is testing using gas chromatograhs nd FIDs. Specifically, this EPA method teaches that VOCs are determined by collecting an approximately 5 gram sample and placing it in a vial with a septum-sealed screw-cap. The vial is sealed and shipped to a laboratory or appropriate analysis site. The entire vial is then placed, unopened, into the instrument carousel. The vial containing the sample is heated to 40 °C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample.

It would have been obvious to one of ordinary skill in the art at the time of the invention to modify the teachings of Robbins in view of Masterton *et al.* and Method AP-42 with the teachings of Turriff *et al.* and/or Method 5035 in order to provide a apparatus as part of the method to obtain a sample size

Application/Control Number: 09/806,274

Art Unit: 2856

between 1 and 100 grams in order that the consistent sample size is placed in the bag prior to sealing.

9. Claim 7 is rejected under 35 U.S.C. 103(a) as being unpatentable over Robbins in view of the teachings of Masterton *et al.* and Method AP-42 as applied to claim 1 above, and further in view of "Determination of Volatile Organic Solvents in Water by Headspace Sampling with the 8200 CX Autosampler" to Penton.

Robbins in view of Masterton *et al.* and Method AP-42 teaches that it is known to provide a sample in a sealed bag (a closed system) and to heat the bag so that the headspace reaches equilibrium. Robbins, in view of Masterton *et al.* and Method AP-42 further teaches that temperature affects the release of VOCs from a material. Robbins in view of Masterton *et al.* and Method AP-42 does not teach a method where the material is held at a temperature between 5 °C and 100 °C.

Penton teaches that it is known in headspace sampling to maintain the closed system, i.e. the 22 mL vial with 10 mL sample, at 80 °C until headspace equilibrium is reached. Again, as known from Masterton *et al.*, equilibrium would have been obtained at any temperature. However more molecules will be released to vapor form at the higher temperature. This will increase the probability of detecting the VOCs in the sample.

It would have been obvious to one of ordinary skill in the art at the time of the invention to modify the teachings of Robbins in view of Masterton *et al.* and Method AP-42 with the teachings of Penton in order to provide a closed

Application/Control Number: 09/806,274

Art Unit: 2856

system (sealed bag) at a temperature between 5 °C and 100 °C in order to reach equilibrium in the headspace.

10. Claims 8-10 are rejected under 35 U.S.C. 103(a) as being unpatentable over Robbins in view of the teachings of Method AP-42, Method 5035, "Manual for the Certification of Laboratories Analyzing Drinking Water - EPA 815-B-97-001" to the EPA, and United States Patent 4,930,906 to Hemphill.

Robbins is a detailed description of the method (instructions) to place samples in a bag so that a) a headspace exits; b) that equilibrium is reached in the headspace; and c) that the headspace is sampled after equilibrium to detect VOCs using an FID. Method AP-42 teaches that systems with mean exit temperature can produce material with VOC emissions. Method 5035 teaches methods (instructions) for obtaining samples of solid materials, placing the samples in a sealed system, maintaining the sealed system at about 40 °C, and subsequently testing the samples for VOCs using a device such as an FID.

EPA-815-B-97-001 specifically requires the need for instructions with a kit used for sampling of VOCs. On page IV-3, section 5 it is stated

"All procedural steps in these methods are considered requirements" Section 6 states

"The manner in which samples are collected and handled is critical to obtaining valid data. It is important that a written sampling protocol with specific sampling instructions be available to and used by sample collectors and available for inspection by the certification officer."

and

Art Unit: 2856

"The sample collector should be trained in sampling procedures and have complete written sampling instructions (SOPs) for each type of sample to be collected."

Furthermore, providing instructions with a sealable bag, thus forming a "kit" is known and is taught by Hemphill. In Hemphill a bag (reference item 10) has instructions (reference items 18 and 20) for temporarily and permanently sealing the bag after material is placed inside.

Providing the bag (from Robbins) and the modified instructions (from Robbins, Method AP-42, and Method 5035) together as a kit (as required by EPA 815-B-97-001) would help ensure that a user, being either a novice or an experienced individual, would be capable of accurately performing the sampling and testing so as to minimize errors that could arise if standard procedures were not followed thus resulting in resampling and retesting.

It would have been obvious to one of ordinary skill in the art at the time of the invention to modify the teachings of Robbins with the teachings of Method AP-42, Method 5035, and Hemphill to provide a kit comprising a sealable bag and instructions for sampling a process system with a mean exit temperature, to place the material in the bag and sealing the bag so that equilibrium is reached at a predetermined temperature, and to test the headspace with an FID in order to detect VOCs.

Conclusion

11. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure.

Art Unit: 2856

a. "Water Quality" to Tchobanoglous shows that the transfer coefficients for several liquids increases with increased temperature.

- b. "A new Method for Field Analysis of Soils Contaminated with Aromatic Hydrocarbon Compounds" to Griffith *et al.* teaches that, in static headspace sampling, a system is closed with a sample inside and that the headspace concentration is related to the initial sample concentration, through thermodynamic equilibrium. It is also taught that the properties of solids and liquids, time, temperature, and volume ratio of headspace to sample all affect the equilibrium distribution of the VOCs.
- c. "A Field Screening Method for Gasoline Contamination Using a Polyethylene Bag Sampling Ssytem" to Robbins *et al.* teaches the basics for which United States Patent 5,140,845 is based. In one discussion the material in the closed system is maintained between 20 °C and 30 °C.
- 12. Any inquiry concerning this communication or earlier communications from the examiner should be directed to David A. Rogers whose telephone number is (571) 272-2205. The examiner can normally be reached on Monday Friday (0730 1600).

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Hezron E. Williams can be reached on (571) 272-2208. The fax phone number for the organization where this application or proceeding is assigned is 703-872-9306.

Art Unit: 2856

Page 15

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dar **&** April 28, 2004

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